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RDX/HMX PLANT DESIGN, PHASE I

JAMES D. TURNER

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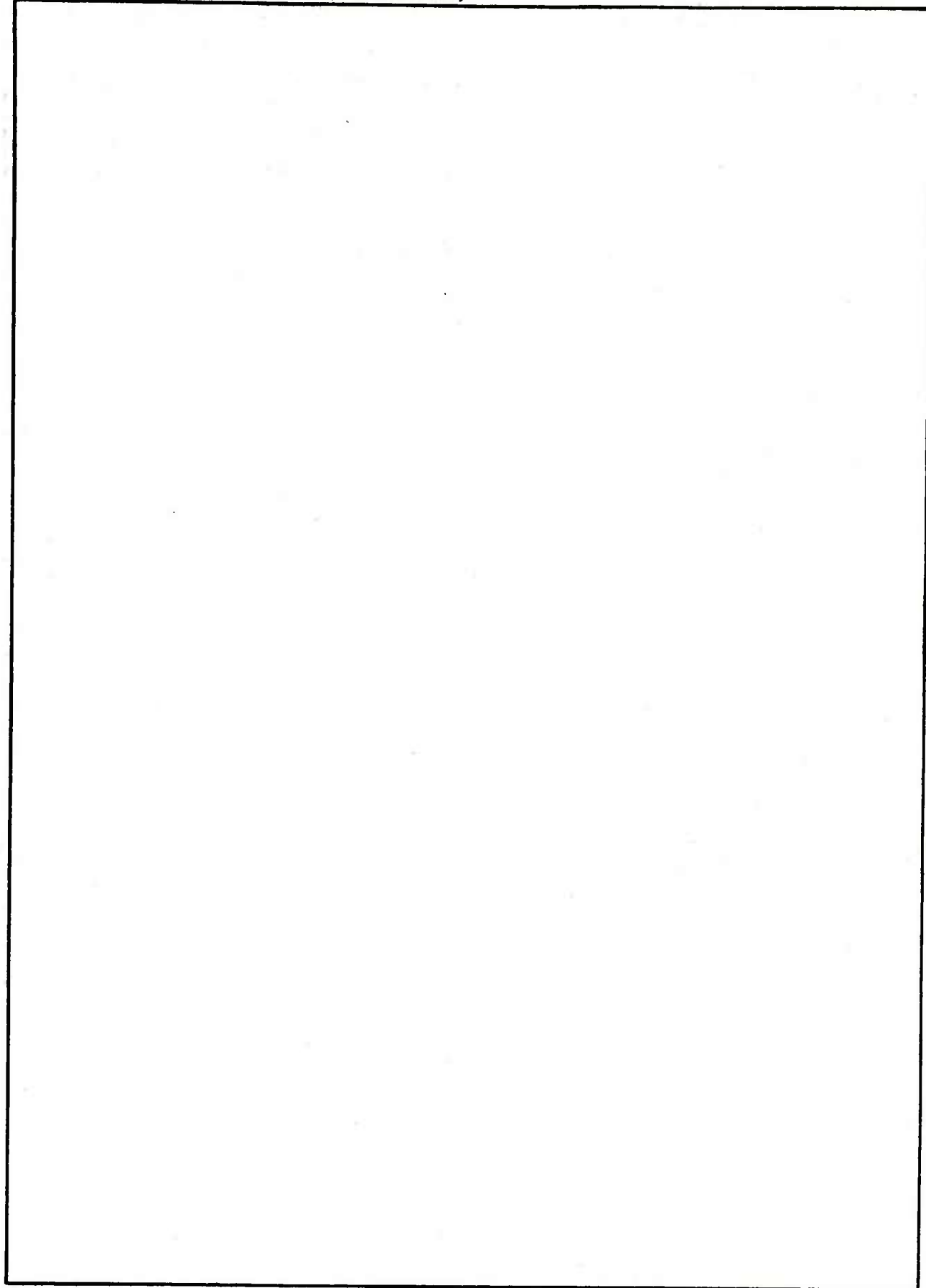
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SUMMARY

The initial design phase of the RDX/HMX Expansion Facility program covered design of nitrolysis, filtration and wash, recrystallization, and wet incorporation and packaging processes on Explosives Manufacturing Line 1; acetic acid distillation, sludge handling, sodium nitrate recovery, ammonia recovery, and acetic acid concentration in the acetic acid recovery area; hexamine/acetic acid solution preparation which prepares raw material for Explosives Manufacturing Lines 1 and 2; and Composition C-4 lacquer preparation which supports the Composition C-4 coating process in Explosives Manufacturing Line 2.

The end products of the initial design effort are process flow diagrams, piping and instrumentation diagrams, motor control schedules, interlock logic diagrams, piping installation drawings, typical instrument installation details, equipment design and specifications, instrument specifications, detailed scale models, operating manuals, hazards analyses, and seismic requirements for the designated areas of Plant X.

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INTRODUCTION

A production capability for the explosives RDX and HMX, and RDX/HMX formulations such as Composition B, Composition C-4, Composition A-5, and Octol exists at only one domestic facility. A program was initiated for expansion of this production base with a second RDX/HMX facility, designated as Plant "X". Plant "X" will accommodate two explosives manufacturing lines with associated supporting material preparation and spent acid recovery areas. Space will be allocated for expanding the facility to four lines. The criteria and designs were prepared under DRC Projects 57X2588 and 57X2688.

DESCRIPTION

Physical Plant

Explosives Manufacturing Line 1 is designed to produce Composition B at an average production rate of 3.4 million kg (7.5 million lb) per month. This production rate is achieved by manufacturing, on a monthly basis, 2.1 million kg (4.5 million lb) of RDX and blending the RDX with 1.4 million kg (3 million lb) of molten TNT and 41,000 kg (90,000 lb) of molten wax.

Explosives Manufacturing Line 2 is designed to produce, on a monthly basis, 2.1 million kg (4.5 million lb) RDX for incorporation into either 3.4 million kg (7.5 million lb) Composition B, or 400,000 kg (900,000 lb) Composition A-5 and 340,000 kg (750,000 lb) Composition C-4, with the balance of the line's RDX capability used for production of 2.2 million kg (4.9 million lb) of Composition B. (Composition B can also be produced on Line 2 at the same rate and in the same manner as on Line 1.) In place of RDX, Line 2 will also be capable of producing 230,000 kg (500,000 lb) HMX to be used in manufacturing 307,000 kg (667,000 lb) Octol 75/25 per month. The recrystallization area of Line 2 will also have sufficient capacity to permit future production of 2.1 million kg (4.5 million lb) per month of Composition A-5 following installation of additional Composition A-5 drying facilities.

Composition C-4 is produced by manufacturing 308,000 kg (679,000 lb) per month of bimodal particle-size distribution RDX and then coating the RDX with lacquer to produce the plastic bonded explosive. Composition A-5 is produced by coating a special granulation of RDX with stearic acid.

Octol 75/25 is produced by manufacturing 230,000 kg (500,000 lb) of trimodal particle-size distribution HMX which is incorporated with 77,000 kg (167,000 lb) of molten TNT per month.

In the nitrolysis area of Lines 1 and 2, RDX and HMX are produced by reaction of the following three feedstreams: hexamine dissolved in glacial acetic acid, ammonium nitrate dissolved in nitric acid, and acetic anhydride. The hexamine is dissolved in acetic acid at the hexamine/acetic acid solution preparation area. The nitric acid/ammonium nitrate solution is produced at the nitric acid/ammonium nitrate preparation area, while the acetic anhydride is produced in the acetic anhydride area.

The acetic acid used in the hexamine/acetic acid solution preparation area and the nitrolysis area is produced in the acetic acid recovery area. A dilution stream of recycled water and acetic acid is also introduced after an initial reaction phase. By varying the raw material ratios, processing temperatures, flow rates, and processing procedures either RDX or HMX can be produced.

Following nitrolysis, the explosive in a spent acid slurry is sent to the filtration and wash area where the spent acid is removed and the explosive is water washed. HMX produced on Line 2 is also purified in this area. The washed explosive is then reslurried in water and pumped to the recrystallization area.

Cyclohexanone and acetone serve as solvents for recrystallization of the RDX and HMX crystals. Recrystallization removes occluded acetic acid, obtains the required particle-size distribution and, in the case of HMX, obtains the more stable beta polymorph. Filtration and screening operations are also used in the Line 2 recrystallization area for the production of different RDX and HMX particle size distributions needed in the manufacture of Composition A-5, Composition C-4, and Octol 75/25. There is also a grinding operation through centrifugal grind pumps to obtain the fine granulation Class 5 HMX, which is blended with other classes of HMX to produce Octol.

Additional support buildings in Line 2 also accomplish blending, weighing, drying, producing of Composition C-4, and packaging.

In the wet incorporation and packaging area, TNT is melted and incorporated with RDX on Lines 1 and 2 or with HMX on Line 2. The RDX/TNT mixture is dried and incorporated with wax to produce Composition B. The Composition B is then cast and packaged. The Octol 75/25 mixture of RDX and TNT is also dried, cast, and packaged. All packaged material is then sent to the loading dock.

In both explosives manufacturing lines, water is recycled with principal losses caused by waste material transported to a Waste Treatment Facility via streams and vapor discharged into the atmosphere. The recrystallization solvents, cyclohexanone and acetone, are recycled with make-up solvent added as required.

Spent acetic acid filtrate, removed in the filtration and wash area, is sent to the acetic acid recovery area to recover acetic acid and residual RDX and HMX.

The acid recovery area consists of the following operations: acetic acid distillation, sludge handling, sodium nitrate recovery, ammonia recovery, and acetic acid concentration.

In the acetic acid distillation operation acetic acid/water filtrate extracted from the explosive slurry in the filtration and wash area of Explosives Manufacturing Lines 1 and 2 is distilled to separate the acetic acid/water from contaminants. RDX and HMX is recovered from the contaminants and the remaining sludge is pumped to the sludge handling operation. The recovered acetic acid is sent to the acetic acid concentration operation.

In the sludge handling operation, sludge from the distillation operation is stripped with steam to remove additional acetic acid which is transferred to the

acetic acid concentration operation. The stripped sludge is causticized, and the resultant sodium nitrate and aqueous ammonia are transferred to the sodium nitrate recovery operation and the ammonia recovery operation, respectively.

Sodium nitrate is recovered in the sodium nitrate recovery operation by vacuum crystallization, centrifuging, and drying. The dried crystalline material is conveyed to the sodium nitrate storage facility prior to being sold.

In the ammonia recovery operation, aqueous ammonia received from the sludge handling operation is treated, and the resultant ammonia is sold.

Vapor phase acetic acid and water is received in the acetic acid concentration operation from the acetic acid distillation and sludge handling operations, and the water content is removed by azeotropic distillation. The resultant glacial acetic acid is distributed to the hexamine/acetic acid solution preparation area, Explosives Manufacturing Lines 1 and 2, and the acetic anhydride area.

The acetic anhydride area, the nitric acid/ammonium nitrate production area, and the waste treatment facility are responsibilities of the United States Army Corps of Engineers.

Basis of Design

The Plant X, RDX/HMX expansion facility design is based on approved criteria documents which represent updating of the basic RDX/HMX technology installed at Holston AAP in terms of process continuity, equipment, materials of construction, process control, instrumentation, etc. The design criteria were modified by Engineering Change Proposals as approved by the United States Army Munitions Production Base Modernization Agency Configuration Control Board. Contractors and Consultants involved in this project are listed in the appendix.

PHASE I DESIGN REQUIREMENTS

The initial authorized Phase I design for Plant X, the RDX/HMX Expansion Facility, covered the design of the process and process equipment including instrumentation for all of Explosives Manufacturing Line 1 processes except for the common TNT receipt and handling building and the common loading dock. These two processes are common to Explosives Manufacturing Lines 1 and 2. Line 1 process designs required were nitrolysis, filtration and wash, recrystallization, and wet incorporation and packaging.

Also included in the design effort was the acetic acid recovery area. This area included a process for acetic acid distillation, sludge handling, sodium nitrate recovery, ammonia recovery, and acetic acid concentration.

Chemical preparation process designs were authorized for hexamine/acetic acid solution preparation which supports Lines 1 and 2 and Composition C-4 lacquer preparation which supports the Composition C-4 coating process located in an adjacent section of the same structure in Line 2.

The scope of work for the design included all direct process operations, equipment design and specifications, instrumentation, and panels, process control systems, and process layout in and out of buildings.

The design of structures, equipment, utilities, and process piping extending 1.5 m (5 ft) beyond the building or area were not included in the scope of work.

Nitrolysis

The nitrolysis process is the first step in the continuous manufacture of Composition B in Explosives Manufacturing Line 1.

The feedstocks for the nitration of hexamine in the production of RDX are pumped to the nitrolysis building from the solution/chemical supply tank farm. All of the feedstreams are provided with return lines to the supply tanks for continuous circulation and temperature stability.

In each of the two reactors, hexamine dissolved in acetic acid is nitrated by a mixture of nitric acid and ammonium nitrate to form crude RDX. The reaction is exothermic. Acetic acid in the hexamine/acetic acid solution acts as a solvent and carrier for the hexamine and does not play an active part in the RDX reaction. The concentrated nitric acid in the nitric acid/ammonium nitrate solution acts as a nitrating agent and as a solvent for ammonium nitrate. An excess of this solution is maintained in the reactors to improve yield.

Acetic anhydride is necessary in the RDX reaction to remove water in the chemical feeds and water generated during the reaction. The acetic anhydride reacts with water to form acetic acid. An excess of acetic anhydride is maintained in each reactor to insure no water is present. This maintains a high RDX yield.

Each reactor is filled with acetic acid from an acetic acid head tank prior to starting the feeds to the reactor.

The major controlled reaction parameters are excess acetic anhydride, excess nitric acid, and reaction temperature. Though the reaction can be controlled over a fairly wide range of temperatures, control of the temperature at 68°C (154°F) produces the highest RDX yield.

Each chemical feedstream to the reactors is controlled by flow rate and ratio station controllers with input from temperature stations for a temperature-corrected feed rate. The hexamine/acetic acid feedstream is the primary reference stream with flow rate controllers of the nitric acid/ammonium nitrate and acetic anhydride streams proportioned to it by ratio stations. The feedstock supply and return streams are equally distributed between two reactor circuits operating in parallel.

In each of the two reactor systems, the hexamine/acetic acid and nitric acid/ammonium nitrate feedstreams are added to the reactor at the reactor pump intake. Acetic anhydride is added through the packing gland of the reactor pumps. Whenever the acetic anhydride is stopped, acetic acid is automatically added to the reactor pump seal to keep explosives flushed from the seal. Chemical additions, along with recirculating anhydrous slurry, are pumped from the reactor pump through a concentric, double-pipe-loop heat exchanger into a reactor vessel. Upon entering the reactor system, the reactants react quickly and become part of the anhydrous RDX/acid slurry. A small portion of this slurry overflows

each reactor vessel into the first of three age tanks in series. The rest is recirculated through the loop heat exchangers. Upon entering the first age tank, the two parallel RDX/acid streams become one. The slurry temperature in each reactor and double-pipe heat exchanger is controlled by the circulation of tempered cooling water through the equipment jacket.

Slurry continuously overflows from the first age tank through the other two age tanks in series. All three tanks are agitated and baffled to improve mixing and cooling. Slurry temperature in the age tanks is kept at 68°C (154°F) (the same as the reactor) by controlling cooling water flow to the tank's bottom and side jackets. The age tanks allow additional time for the reaction to go to completion, which improves RDX yield.

The overall yield is approximately 2.6 kg (5.7 lb) of RDX per kg of hexamine.

From the last age tank, the slurry overflows into the first of two agitated simmer tanks installed in series. During the simmering operation, the simmer tanks are vented to a contact condenser system and the condensate, combined with scrubbing water, is returned to the first simmer tank as an aqueous dilution liquor. This dilution liquor, consisting of 10% \pm 5% acetic acid and water, is added to the first simmer tank to maintain an acetic acid concentration of 63% \pm 2% in the simmer system. This liquid is added to the system to hydrolyze undesirable by-products of the RDX reaction. The slurry temperature in the simmer tanks is maintained at 109°C (226°F) to speed up the hydrolysis of by-products.

A nitrogen purge system is provided for the simmer tanks to prevent the possibility of having a flammable mixture of hot acetic acid and air during startup or shutdown of the process. The hot simmer tanks are purged automatically by a positive set pressure being maintained in the simmer tank vent to the contact condenser.

The hot slurry overflows from the first to the second simmer tank and then into the first of five jacketed and agitated cooling tanks. At the first tank, the temperature is lowered to 98°C (209°F). The slurry overflows continuously from one cooling tank to the next and is cooled gradually through each succeeding tank from 98°C to 50°C (209°F to 122°F). From the cooling system, the RDX slurry is pumped through a heat exchanger which further cools the slurry to 40°C (104°F). The slurry is cooled to promote almost complete precipitation of the RDX in preparation for filtration in the filtration and wash building. Following the heat exchanger, the RDX slurry is piped to the filtration and wash building.

Filtration and Wash

The filtration and wash process is designed for continuous filtration and washing of crude RDX/acetic acid slurry received from the nitrolysis building to remove and recover acid filtrate and to supply RDX slurry of low acid content to the recrystallization building for processing.

Crude RDX slurry is pumped from the nitrolysis building to an agitated slurry feed tank in the filtration and wash building. The crude RDX slurry is pumped from the slurry feed tank to a continuous belt filter. Filtration is accomplished in three belt filter vacuum zones.

In the first zone, the slurry is filtered to remove and recover acetic acid filtrate. The filtrate flows to a vacuum receiver from where it is pumped to either of two acid filtrate storage tanks located at the filtration and wash building. RDX fines which pass through the filter and settle in the acid filtrate storage tanks, as well as additional RDX recovered in the acetic acid recovery area, are periodically pumped to a recovered RDX tank. This recovered RDX is blended with the RDX from the main nitrolysis building to accomplish recovery.

In the second zone, the RDX is washed with water from the building scrubber. The resulting filtrate flows to a dilution liquor vacuum receiver. Dilution liquor is then pumped first to an intermediate storage tank and then to the nitrolysis building for the scrubbing and condensation of hot simmer tank vent gases.

In the third zone, the RDX cake is washed two additional times in two process water streams. This wash water stream flows to a vacuum receiver and the liquid is then added to the vacuum seal water loop and used to wash the revolving belt of the belt filter.

Reslurry water supplied from the recrystallization building is used as sluice water to move the wet RDX cake off the filter to a reslurry tank. The filtered RDX is reslurried with additional reslurry water and is pumped to the recrystallization building.

Recrystallization

Washed, crude RDX slurry is received by pipeline from the filtration and wash building and the RDX is partially dissolved in the solvent cyclohexanone. The solvent is then distilled and recovered for reuse, leaving recrystallized RDX in a water slurry. The RDX is recrystallized to conform to acceptable tolerances and specifications. The RDX is then pumped to the wet incorporation and packaging building. While the receipt and delivery of RDX slurry to and from the recrystallization building is a continuous operation, the recrystallization process is accomplished under a batch mode.

RDX slurry, pumped to the recrystallization building, is received in a jacketed and agitated slurry surge tank. The slurry is pumped from the surge tank sequentially, in measured quantities, into one of five jacketed and agitated recrystallizers. Proportional amounts of preheated cyclohexanone are pumped from a cyclohexanone storage tank to the recrystallizer. Filtrate water is used for batch-weight adjustments and slurry line flooding and flushing.

Dissolution of RDX in cyclohexanone, cyclohexanone distillation, RDX recrystallization, and RDX/water slurry cooling is accomplished in the crystallizers. Agitators are used to hold solids in suspension and to accelerate dissolution and evaporation. The crystallizers are equipped with jackets, coils, and sparger steam for heating and cooling requirements of the recrystallization process. Nitrogen is also introduced through the sparger pipe to reduce noise and provide an inert atmosphere. When the recrystallization cycle is complete, the RDX slurry is pumped to a holding tank. From the holding tank, the RDX is transferred by pipeline to the wet incorporation and packaging building.

Wet Incorporation and Packaging

RDX/water slurry received from the recrystallization building is dewatered, weighed, conveyed to an area where it is incorporated with molten TNT, dried, and mixed with molten wax to form Composition B. The molten Composition B is solidified, flaked, and packaged for storage or shipment.

Cooled RDX/water slurry pumped from the recrystallization building is received in a slurry feed tank and pumped to a continuous belt filter for vacuum-induced dewatering. A vacuum system consisting of a vacuum pump and a receiver is provided to accelerate dewatering. Through filtration, the water content of RDX is reduced from 6% to 10% of the total weight. The flow of the feed to the filter is controlled with slurry density corrections incorporated. A vacuum pump seal-water system with a tank and pumps is provided. A heat exchanger is also provided to cool the vacuum seal water.

Filtrate from dewatering is passed through a vacuum receiver and collected in a filtrate tank. A portion is pumped to the belt filter for filter belt assembly washing and the remainder is pumped to the recrystallization building. Water and RDX recovered from the filter wash is collected in the reclaimed water tank and pumped to the belt filter to recover the RDX.

Agitators and pump seals are supplied with a fixed amount of seal water from a seal water loop. This system consists of a tank, pumps, an exchanger for heat removal, and a filter.

Full TNT transfer bins from the TNT receipt and handling building are received on an interbuilding conveyor. The full transfer bins are received and accumulated on an accumulating conveyor. The accumulation of four full bins stops the interbuilding conveyor and restarts it when only two full bins remain. The interbuilding conveyor controls the feed of TNT bins to the conveyor to maintain a minimum 40 m (130 ft) spacing between full bins at all times. The spacing is required to avoid explosive propagation from one bin to the next.

From the accumulating conveyor, the bins of TNT are dumped into a TNT hopper. When the level of TNT in the hopper is low enough to receive the contents of a transfer bin, a singulator releases a bin from the accumulating conveyor onto a shuttle conveyor. The conveyor moves the bin into the arm of a bin inverter which empties the contents into the TNT hopper. The TNT hopper has high and low level indicators with an interlock to the bin inverter.

The empty bin is then lowered to the weighing conveyor. The empty TNT bin weight is checked, and any partially unloaded bins are diverted onto a storage section for subsequent manual unloading. Empty bins are moved to the lower section of the two tiered, interbuilding conveyor system, which runs between the wet incorporation and packaging building and the TNT receipt and handling building. These lower, belt-type conveyors have powered rollers at all turning points and are completely enclosed for protection against inclement weather. Since the bins are empty, there are no spacing requirements on the lower tier of this system.

The RDX weigh feeder continuously receives and weighs the wet RDX from the belt filter and transfers it into the incorporation kettle. The TNT weigh feeder receives and weighs TNT from the TNT hopper and continuously feeds it to the TNT

melter. The TNT feed rate is controlled by the RDX weigh feeder. Molten TNT overflows from the melter into the incorporation kettle. The homogeneous mixture of molten TNT and RDX overflows the incorporation kettle into a decant tank, where water separates from the wet RDX and overflows from the decant tank into a catch tank.

The catch tank provides sufficient residence time so that any explosive material carried from the decanter by the water will settle. The catch tank liquid overflows into a quench tank and is pumped from the quench tank through a heat exchanger into the catch basin.

When an accumulation of explosives in the catch tank is detected, the agitator starts, and the explosive is slurried and transferred to an empty separator tank. The explosive is allowed to settle and the concentrated explosive is transferred to the decanter. The water phase is then transferred to the quench tank.

The molten mixture of TNT, RDX, and remaining water overflows from the decanter to the first of three heated tank dryers arranged in series. The vapors from the drying kettles are processed through a scrubber to condense the water, TNT, and cyclohexanone vapors. The molten mixture overflows the last of the three dryers into a kettle to which wax is added to produce molten Composition B. The molten Composition B overflows the waxing kettle onto the casting belt.

Molten Composition B is received continuously by the casting belt flowing into a steam-heated casting bar. This assembly consists of a heated stainless steel casting bar with unheated stainless steel side dams and an unheated polyethylene rear dam. The stainless steel casting bar is fitted with a replaceable notched polyethylene bar at the bottom. The notches control the product strip thickness and width. Composition B is cast into a 1.5 m (60 in.) wide, 1.5 mm (1/16 in.) thick strip with notches to facilitate subsequent strip breakage at the discharge roll. Chilled water is sprayed onto the underside of the casting belt to help solidify the strips which are broken into approximately 10 mm (0.4 in.) squares (flakes) by a scraper blade at the end of the belt discharge.

The casting belt is provided with an air hood assembly to exhaust the air above the Composition B. The casting belt has a steel structure to support its entire length and an automatic speed control with a remote controller.

A recirculating chilled water system is provided to cool the water that is sprayed on the underside of the casting belt.

From the end of the casting belt, the solidified Composition B falls into the feed hopper of a vibrating feeder. The feeder is equipped with a full dustcover and is driven by an air motor using rubber isolation mounts. It operates at a full flow rate and a dribble flow rate to fine trim each 27 kg (60 lb) batch. The point of change between full flow and dribble flow is adjustable. The vibrating feeder discharges into a bagging scale which is also equipped with full dustcover and is connected to the vibrating feeder by an electrically conductive flexible sleeve. The function of the bagging scale is to weigh and place 27 kg batches of Composition B into liners. Following discharge, the hopper discharge valve closes and a new cycle is initiated.

A carton-forming machine constructs a corrugated carton, tapes the bottom flaps closed, and places it on a conveyor. A dust collection enclosure is provided at the carton former to collect any explosive dust on any of the used boxes.

Empty cartons are received from the carton-forming machine and accumulated on a gravity-type accumulation conveyor. A maximum of eight empty cartons can accumulate on this conveyor prior to proceeding to the bagging scale for loading. Position or limit switches are provided to stop the transfer of cartons when the conveyor is full and restart the transfer when it is low. A singulator releases the cartons, one at a time, onto the indexing conveyor when the bagging scale is ready to accept a carton.

The indexing conveyor is a power-driven conveyor that positions empty cartons under the bagging scale. After a liner filled with 27 kg (60 lb) of Composition B drops into the carton, the conveyor transfers the filled, unsealed carton to a transfer conveyor. This powered conveyor transports filled unsealed cartons to the conveyor and carton shaker. This unit is a vibrating-type conveyor that moves the cartons through a metal trough with an oscillating motion. As the filled cartons transverse along the conveyor, the Composition B settles into the carton liner. A dust collection hood is provided over the conveyor to keep the dust out of the operating area.

Moving along the conveyor, the cartons proceed through the liner sealer, carton closer, and coder units. This system operates as an integral part of the conveyor system. The liner sealer unit creases, folds, and pushes the liner into the carton. The carton closer unit closes and tapes the one-piece box with a series of cam-operated arms and guides. The coder unit applies a 2.5 x 30 cm (1 x 12 in.) strip of printable pressure-sensitive tape to the side of the carton and prints the required identification on it. All Composition B packaging operations are automatic with manual by-pass capabilities. The closed coded cartons are next conveyed onto an accumulation conveyor. This conveyor is a powered belt unit that accumulates and conveys the cartons to an interbuilding conveyor. The accumulating conveyor can also be used to accumulate cartons for manual palletizing in the building. The interbuilding conveyor consists of powered belt sections with powered rollers at all curves. This conveyor transports filled closed cartons of Composition B to the loading dock. The belt maintains speeds that ensure a 7.5 m (25 ft spacing) between cartons to avoid explosive propagation from one box to the next in the event of an accidental detonation.

Acetic Acid Recovery Area

The acetic acid recovery area is designed to receive and process the acid filtrate extracted from the filtration and wash areas of Explosives Manufacturing Lines 1 and 2 and the furnace acid extracted from the acetic anhydride process area. This area is comprised of the processes discussed below.

Acetic Acid Distillation

The acetic acid to be recovered is received at the acetic acid distillation operation in either of two filtrate receivers. The filtrate receivers are heated to maintain a minimum filtrate temperature of 45°C (113°F), thus preventing the dissolved RDX from settling out of solution and depositing on the tank

bottom. In addition, the vessels are equipped with agitators to further prevent the settling and deposition of undissolved solids. Solids that may deposit in the transfer lines are removed periodically by flushing the lines.

Weak acetic acid (furnace acid) produced as a by-product of acetic anhydride manufacture in the acetic anhydride area is also received and processed in the acetic acid distillation area. Prior to mixing the furnace acid with the acid filtrate, coke is removed from the furnace acid through filtration and the furnace acid is pumped into a heated furnace-acid receiving tank. A low boiler is still used to remove acetone and methyl acetate before the furnace acid is mixed with acid filtrate for further processing. The acetone and methyl acetate are condensed and pumped to the waste disposal area.

Following mixing of the acid filtrate and furnace acid in the filtrate receivers, the filtrate is mixed with a 50% sodium hydroxide solution in an in-line mixer to neutralize the nitric acid content. Acid neutralization is controlled by a flow element and transmitter in the filtrate transfer line which ratios filtrate and sodium hydroxide flow to the mixer.

The resulting filtrate is pumped to either of two filtrate test tanks. The filtrate test tanks are heated to 45°C (113°F) to maintain dissolved RDX in solution. In addition, the tanks are equipped with agitators to further prevent the settling and deposition of undissolved solids on the tank bottom.

Treated filtrate is pumped from the test tanks through a heater to the primary evaporator where approximately 85% of the water and acetic acid content is vaporized and removed overhead to a primary separator. The separator prevents carry-over of entrained solids and liquids in the acetic acid/water vapor stream from the evaporator. From the primary separator the vapor flows to the azeotropic column in the acetic acid concentration area.

The concentrated or bottom product from the primary evaporator, containing approximately 1.5% RDX in a solution of water, acetic acid, and nitrate salts, is pumped to one of three crystallizers to recover the RDX.

The three crystallizers are charged in sequence with the evaporator bottom product and diluted with water supplied from the nitric acid manufacturing area. The small amount of nitric acid in the dilution water is neutralized by the excess sodium hydroxide carried through from the initial neutralization process.

A nominal amount of recovered RDX slurry is extracted from batches of previously recrystallized RDX. This is reinjected sequentially in each crystallizer as seed material to initiate crystallization. Each crystallizer is then cooled to reduce the RDX solubility resulting in the precipitation of the RDX.

To effect the required cooling, the crystallizers are provided with both cooling water to initiate the cooling and chilled water for reaching the final crystallization temperature. The slurry content of each crystallizer is pumped to a cyclone separator which concentrates the recovered RDX. The concentrated RDX is then transferred to a slurry receiver tank, and the separated liquor is transferred to the secondary evaporator feed tank.

The RDX is reslurried in the slurry receiver by the addition of fresh water which reduces the RDX concentration to approximately 20%. The slurry is then pumped to the filtration and wash building.

The liquid in the secondary feed tank is pumped from the tank to the evaporators. One half is distributed to the primary evaporator for dilution of the acid filtrate stream to maximize the fraction evaporated. The balance is sent to the secondary evaporator where additional extraction of water and acetic acid occurs.

The resulting vapors from the secondary evaporator are discharged to a secondary separator to remove entrained solids and liquid and provide vapor product to the acetic acid concentration process. The secondary evaporator bottom concentrate is pumped to the sludge/handling process.

Sludge Handling

The concentrated sludge discharged from the bottom of the secondary evaporator in the acetic anhydride distillation process is received in the stripper feed tank of the sludge handling process where the RDX content is retained in solution and suspension by a heater. The concentrate is pumped from the stripper feed tank to the sludge stripper. Stripping action is induced by steam injected into the bottom of the stripper. The acetic acid is stripped from the solution along with water vapor, which is sent to the acetic acid concentration area.

The stripper bottom sludge (approximately 65% nitrate salts) flows to a surge tank where it is reacted with a 50% sodium hydroxide solution from a caustic soda day tank. The residual RDX in the sludge is hydrolyzed during causticization, and the sodium hydroxide reacts with the ammonium nitrate salt to produce additional sodium nitrate and ammonia vapor. The sodium nitrate solution is pumped sequentially from the causticizers to the sodium nitrate recovery area.

The ammonia vapor is vented to an ammonia scrubber where it is scrubbed with recycle water from the ammonia recovery area. The resultant 8.3% aqueous ammonia solution is discharged to the ammonia recovery area. Scrubber temperature is controlled by an ammonia scrubber cooler.

The caustic soda day tank also supplies sodium hydroxide solution to the acetic acid distillation area and the ammonia recovery area.

Acetic Acid Concentration

The vapor phase feedstock from the acetic acid distillation process is fed directly to an azeotropic still where removal of the water and the contaminants is accomplished by the azeotropic agent n-propyl acetate. The azeotropic agent supply is maintained by means of a solvent recovery loop which is supplemented by make-up from an n-propyl acetate storage tank. The tank has the capability of storing n-propyl acetate from the system and supplying the system during startup with sufficient agent to accomplish the process. Heat input to the azeotropic still is accomplished by means of a thermosiphon reboiler.

The bottoms product, consisting of glacial acetic acid, is cooled to 40°C (104°F) in a heat exchanger and pumped to one of two glacial acetic acid

storage tanks. During startup, the storage tanks may be bypassed and the product discharged to an off-spec acetic acid tank. The off-spec product is pumped back to the still at a low rate for recovery after product purity has been obtained in the still. The high-purity product retained in the storage tanks is distributed for use in the acetic anhydride manufacturing plant, the hexamine/acetic acid solution preparation area, and in the nitrolysis area. The recovery of glacial acetic acid from the feedstock is supplemented by purchased acetic acid to satisfy the plant demands. The purchased acid is pumped to the glacial acetic acid storage tanks from an unloading station.

The azeotropic still overhead vapor containing n-propyl acetate, water, and various products of reaction is condensed, cooled to 75°C (167°F), and discharged to a hot azeotropic decanter for separation into two liquid phases. The decanter is provided with a vent condenser to recover solvent vapors. Operation of the hot azeotropic decanter at 75°C (167°F) minimizes the concentration of organics and solvent in the water underflow product.

The underflow from the azeotropic decanter is pumped through the stripper sweet water and slop water exchanger where it is preheated and fed to the sweet water stripper. The hot stripper bottoms are pumped to the heat exchanger as the heating medium for the stripper feed. The stripper uses steam injection at 100 kPa (15 psig) to heat the product and strip the light end contaminants. The bottoms product, cooled in the stripper sweet water and slop water exchanger, is discharged to waste treatment for disposal. The overhead product is condensed in the sweet water stripper condenser and returned to the hot azeotropic decanter.

The overflow product from the azeotropic decanter containing approximately 73% n-propyl acetate, is pumped through a heat exchanger which uses the underflow product of the cold decanter as a cooling agent. The product then passes through a second azeotropic agent cooler which uses cooling tower water as the coolant. The final stage of cooling is through an azeotropic agent chiller which uses cooling tower makeup water as the cooling agent to reduce the product temperature to 25°C (77°F). The liquid then enters the cold azeotropic decanter which operates at 25°C (77°F) to reduce the solubility of water in the n-propyl acetate. The underflow product, containing approximately 94% water, is pumped through the azeotropic agent heat exchanger and back to the hot azeotropic decanter. The overflow product, containing approximately 75% n-propyl acetate, is pumped back to the azeotropic still as reflux. A portion of this overflow stream is removed as a purge and fed to the purge still.

A continuous purge of the recovered n-propyl acetate is provided to stabilize the concentration operation by providing constant quality solvent, improve operating economics by eliminating the periodic total replacement of the solvent, and to reduce the loss of n-propyl acetate when purging propyl formate from the product. The purge stream is fed to a purge still where the heat for distillation is supplied by a thermosiphon reboiler. The purge still reduces the level of contamination in the n-propyl acetate bottoms product to approximately 7% from a level of approximately 25% in the feedstream. The bottoms product is pumped back to the azeotropic still solvent feedstream for reuse in the system. The overhead vapor is condensed and cooled to 40°C (104°F) in the purge still condenser and discharged to the purge still decanter. The decanter overflow is partially refluxed to the purge still and partially discharges from the system to

the waste treatment facility as a continuous purge of the contaminants. The underflow is pumped intermittently to the hot azeotropic decanter as required. Excess flow is discarded for waste treatment and disposal.

Nitrogen blanketing is provided to preclude a potentially explosive atmosphere in the n-propyl acetate storage tank, the hot azeotropic decanter, the cold azeotropic decanter, and the purge still decanter.

Sodium Nitrate Recovery

The sodium nitrate feed tank receives 49% sodium nitrate solution from the sludge handling facilities. The solution contains small quantities of sodium hydroxide (0.9%), sodium formate (4.9%), and sodium acetate (2.1%). The nitric acid manufacturing facility supplies a 60% nitric acid solution to the nitric acid feed tank.

Sodium nitrate feed pumps and nitric acid feed pumps feed the respective solutions to an in-line mixer. A sufficient quantity of 60% nitric acid is mixed with the sodium nitrate to convert the bulk of the residual sodium hydroxide to sodium nitrate. The mixture is then discharged to a recycle mix tank. In this tank, the mixture is blended with a more dilute stream of previously processed solution that is returned for recycling. The solution is then pumped to the evaporator-crystallizer where it is concentrated to 60% sodium nitrate by overhead removal of 49% of the water. The sodium nitrate is partially crystallized in the evaporation process.

Water vapor taken overhead from the evaporator-crystallizer is condensed in the evaporator condenser and discharged as hot water to the hot well tank. Two steam jet eductors with jet condensers are used to exhaust the evaporator-crystallizer and maintain a vacuum of 50 mm Hg (abs). The condensate is drained to the hot well tank and mixed with the condensate from the evaporator-crystallizer. The clean condensate from the hot well tank is used as makeup water in two vent scrubbers.

The slurry extracted from the bottom of the evaporator-crystallizer is pumped to a centrifuge where a 91.5% concentrate of sodium nitrate is separated and discharged to the high-concentration sodium nitrate dryer. The dryer removes residual water from the concentrate by the counterflow of preheated air. An air and water-vapor mixture is discharged to a scrubber where it is scrubbed free of solid particles and discharged to the atmosphere. The scrubber water bottoms are returned to the recycle mix tank. The dry high-concentration (96%) sodium nitrate is removed from the dryer and conveyed to the sodium nitrate storage facility.

The low-concentration sodium nitrate solution is pumped and sprayed onto a recycle loop of dry low-concentration sodium nitrate. This stream is then sent to the dryer. Drying is accomplished by an airstream preheated with steam. The dryer recycles the 71% sodium nitrate product through the recycle loop using screw conveyors on a 10-to-1 ratio (recycle to discharge) to minimize dusting and clogging of the equipment. Extracted water vapor carried by the airstream is passed through a second scrubber where entrained solids are washed from the stream and pumped back to the surge sodium nitrate tank for recycling through the low-concentration sodium nitrate dryer.

Parallel screw conveyors for high-concentration and low-concentration material receive sodium nitrate products through a chute from the dryers and convey them to bucket elevators and conveyors, which transport the products to their appropriate storage areas in the warehouse.

Ammonia Recovery

The 8.3% aqueous ammonia solution produced in the sludge handling area is received in the aqueous ammonia feed tank. The solution is pumped to an in-line mixer which blends the solution with 20% sodium hydroxide solution supplied from a feed tank through metering pumps. The blended solution is preheated and discharged to an ammonia still. Blending converts residual carbon dioxide to sodium bicarbonate for elimination in the still bottoms.

Overhead vapors discharged from the still contain approximately 25% methylamines and 75% ammonia. The vapors are condensed in the still vent condenser and discharged to an ammonia receiver tank. The ammonia pumped from the receiver tank is split into two streams. A portion is returned to the still as reflux, and the remainder is discharged to the ammonia-amine test tank. Product exhibiting excess water is returned through an in-line mixer (where plant water is added) to the aqueous ammonia feed tank for recycling through the still. Acceptable product is pumped to a tank car loading facility.

The still bottoms temperature is maintained by circulating the still bottoms through a reboiler. The discharge from the still bottom consists primarily of water with approximately 3% hexamine. This hot liquid is discharged through two parallel cooling heat exchangers. A portion of the still bottoms is recycled to the ammonia scrubber in the sodium nitrate recovery area while the remainder is diverted to the waste treatment facility.

Hexamine/Acetic Acid Solution Preparation

To produce a batch of hexamine/acetic acid solution, acetic acid is pumped into a dissolver vessel until a quantity sufficient to cover the agitator has been reached. Hexamine is then introduced into the dissolver vessel from a weigh feeder. Acetic acid feed also continues until the required quantity has been added. The batch preparation process lasts approximately 190 minutes plus an additional 20 minutes for transfer to storage.

Hexamine is transferred to the dissolver by means of a mechanical conveying and weighing system. Dust is collected in drums and removed from the premises. Glacial acetic acid is pumped from an acetic acid day tank and metered to the dissolver.

The dissolver tank is agitated to promote and accelerate dissolution. Cooling coils are provided to remove the heat of dissolution. The coils are also used, with steam, to maintain minimum temperature and to prevent recrystallization of hexamine.

After batch completion, the hexamine/acetic acid solution is pumped to the solution and chemical supply area. An 8-mesh screen is provided on the dissolver outlet to prevent foreign matter from reaching the densitometer or production lines.

Composition C-4 Lacquer Preparation

Composition C-4 is a plastic bonded explosive prepared by coating a blend of RDX of two specific particle size distributions known as RCA-2 and RDX Class 5 with lacquer and drying the resultant product. In the lacquer preparation process, the solid elastomer polymer is chopped into small pieces and dispersed into toluene. Dioctyl adipate and process oil are added and dissolution is promoted by heat and agitation.

To prepare a batch of lacquer, the required amount of toluene is pumped from the toluene storage tank to the lacquer mixer. Palletized loads of elastomer are delivered from storage by forklift truck, manually unpacked, and loaded on a conveyor. Blocks of elastomer elevated by the conveyor are fed through a hopper to a chopper which reduces the elastomer to 1 cm (0.39 in. or smaller) pieces which drop onto a weigh feeder. The weigh feeder weighs, totalizes, and discharges the required batch size of elastomer to the lacquer mixer which previously received the toluene. Dioctyl adipate is then pumped and metered to the lacquer mixer from a dioctyl adipate storage tank. The mixture is agitated, heated to 70°C (158°F), and checked for undissolved solids. Process oil is pumped from the process oil tank and metered to the mixer. The lacquer is mixed and then pumped to the C-4 production process.

The lacquer mixer is provided with an agitator and is jacketed for steam heating (and cooling, if required) to promote dissolution of the elastomer. Nitrogen blanketing is provided to prevent the formation of an explosive vapor-air mixture in the vessel. Condensing and refluxing of vapors is accomplished by a chilled water vent condenser.

CONCLUSIONS

Phase I of the Plant X, RDX/HMX Expansion Facility design of Line 1 (Comp B) and Acetic Acid Recovery-Concentration Areas proved feasible.

Design data and basic equipment specifications from Line 1 are applicable for the design of multi-product Line 2.

A systems engineering approach proved to be an excellent management tool for integrating the many tasks associated with the program and for controlling the DARCOM Corps of Engineers interfaces.

RECOMMENDATIONS

It is recommended that design of Plant X, RDX/HMX Expansion Facility be completed, followed by construction, installation, and operation of the facility. This will increase RDX and HMX production capacity while also providing the strategic value of providing an alternate site of manufacture.

APPENDIX

CONTRACTOR, SUBCONTRACTORS, AND CONSULTANTS

The following contractor, subcontractors, and consultants participated in the design of the process:

Ralph M. Parsons Company

Ralph M. Parsons Company, Pasadena, California 91124, performed as contractor for the detailed design of this project. Parsons was responsible for preparing flow diagrams, piping and instrumentation diagrams, motor control schedules, interlock logic diagrams, piping installation drawings, typical instrument installation details, equipment designs and specifications, instrument specifications, operating manuals, hazards analyses, and seismic requirements.

A. T. Kearney, Incorporated

A. T. Kearney, Inc., 100 South Wacker Drive, Chicago, Illinois, 60606, performed as a subcontractor of Ralph M. Parsons to provide a summary of applicable health and safety standards, and to participate in the review of Ralph M. Parsons' prepared process flow diagrams, piping and instrumentation diagrams, equipment procurement specifications, scale models, and operating manuals for materials handling systems.

Allegheny Ballistics Laboratory of Hercules, Incorporated

Allegheny Ballistics Laboratory located in Cumberland, Maryland, 21502, performed as a subcontractor of Ralph M. Parsons to provide the hazards analysis and loss prevention study for each of the process design areas.

Hercules, Incorporated

Hercules, Inc., 910 Market Street, Wilmington, Delaware, 19899, performed as a subcontractor to Ralph M. Parsons on the initial phase of the design for nitrolysis, filtration and wash, acetic acid distillation, sludge handling, sodium nitrate recovery, ammonia recovery, hexamine/acetic acid solution preparation, and Composition C-4 lacquer preparation.

Hercules, Inc. also monitored Holston AAP's RDX and HMX process technology, reviewed the progress and final reports of manufacturing methods and technology engineering funded projects covering RDX and HMX related processes, and reviewed process and process equipment documents prepared by Parsons for completeness and accuracy.

Maurice Baer

Maurice Baer, 47 Dacotah Avenue, Rockaway, New Jersey, 07866, assisted Ralph M. Parsons Company as a consultant. He participated in the review of all design documents prepared.

Norman L. McLeod

Norman L. McLeod, 3252 Teton Drive, Salt Lake City UT, 84109 assisted the Ralph M. Parsons Company as a consultant. He participated in the review of operations and layaway manuals for format and content.

John A. Ruth

John Ruth, 2119 Swinnen Drive, Wilmington, Delaware, 19803, assisted Ralph M. Parsons Company as required for specific process studies.

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